



PATENT  
Serial No. 10/797,301 (DE258-68395/DP-308342)  
Declaration Under 37 C.F.R. § 1.131

## IN THE UNITED STATES PATENT & TRADEMARK OFFICE

Applicant(s): Robert J. Svoboda et al.	)	Examiner: S. Ahmed
	)	
Serial No.: 10/797,301	)	Art Unit: 1792
	)	
Filed: March 9, 2004	)	Confirmation No. 4465
	)	
For: CERAMIC ASSEMBLY WITH A	)	
STABILIZER LAYER	)	
	)	

### ROBERT J. SVOBODA – DECLARATION UNDER 37 C.F.R. § 1.131

**Mail Stop Amendment**  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

I, Robert J. Svoboda, declare that:

1. I was employed as a contract employee with Delphi Technologies, Inc. ("Delphi"), Troy, Michigan, during the relevant time period.
2. I graduated with a B.S. degree in chemical engineering from Michigan State University.
3. I am one of the inventors identified in the above-identified U.S. Patent Application Serial No. 10/797,301, filed on March 9, 2004, for a Ceramic Assembly With A Stabilizer Layer.

4. On or before August 1, 2002, Joseph M. Keller, another co-inventor in above-identified U.S. Patent Application Serial No. 10/797,301, and I conceived a method of producing a ceramic assembly, comprising: disposing an electrode precursor on an electrolyte precursor having an electrolyte sintering shrinkage; disposing a stabilizer precursor having a stabilizer sintering shrinkage on the electrode precursor on a side opposite the electrolyte precursor to form a precursor assembly, wherein a difference between the electrolyte sintering shrinkage and the stabilizer sintering shrinkage is less than or equal to  $\pm 1\%$ ; and sintering the precursor assembly to form the ceramic assembly comprising a stabilizer layer, electrode, and electrolyte, wherein a surface of the ceramic assembly has less than or equal to about 5.0 degrees camber, as measured from the horizontal plane. See *U.S. Patent Application No. 10/797,301*, claim 1.

5. On or before August 1, 2002, Joseph M. Keller and I conceived a method of producing a solid oxide fuel cell, comprising: disposing a first electrode precursor on a solid electrolyte precursor having an electrolyte sintering shrinkage; disposing a stabilizer precursor having a stabilizer sintering shrinkage on the first electrode precursor on a side opposite the electrolyte precursor to form a precursor assembly, wherein a difference between the electrolyte sintering shrinkage and the stabilizer sintering shrinkage is less than or equal to  $\pm 5\%$ ; disposing a second electrode precursor on a side of the solid electrolyte precursor opposite the first electrode precursor; and sintering the precursor assembly to form the ceramic assembly comprising a stabilizer layer, first electrode, and electrolyte, wherein a surface of the ceramic assembly has less than or equal to about 5.0 degrees camber, as measured from the horizontal plane. See *U.S. Patent Application No. 10/797,301*, claim 26.

6. Attached hereto is a true copy of relevant portions of a record of invention that was prepared in the United States of America prior to August 1, 2002 describing a method of adding a stabilizing layer to a solid-oxide fuel cell to increase the flatness of the solid oxide fuel cell, which was signed by Haskell Simpkins, Joseph M. Keller and me prior to August 1, 2002. *See Exhibit A (redacted)*, 7 pages.

7. The record of invention referred to in Paragraph 6 includes a description of a method of producing a ceramic assembly or solid oxide fuel cell including the steps of providing a electrolyte-anode precursor composite bi-layer, and disposing a stabilizing precursor layer on an anode side of the electrolyte-anode precursor composite bi-layer, wherein the stabilizing precursor layer has similar shrinkage properties as the electrolyte precursor to counter-balance the distortion caused by the shrinkage mismatch of the electrolyte and the anode that occurs during a sintering operation. *See Exhibit A (redacted)*, pgs. 1-3.

8. The record of invention referred to in Paragraph 6 shows that the method includes disposing a cathode precursor on a side of the electrolyte precursor opposite of the anode precursor. *See Exhibit A (redacted)*, pg. 3.

9. The record of invention referred to in Paragraph 6 discloses an illustration of the ceramic assembly or solid oxide fuel cell that is formed using the method described in Paragraphs 7 and 8, which includes a stabilizing layer, an anode, an electrolyte, and a cathode. *See Exhibit A (redacted)*, pg. 3.

10. Attached hereto as Exhibit B is a true and original copy of a presentation entitled "Addition of Stabilizing Layer to Increase Solid Oxide Fuel Cell Flatness Invention No. DP-308342" that Joseph M. Keller, Haskell Simpkins and I prepared on or before August 1, 2002 in the United States of America. *See Exhibit B*, 8 pages.

11. The presentation referred to in Paragraph 10 discloses a solid oxide fuel cell including a first electrode (i.e., anode and active anode) disposed on an electrolyte, and a stabilizing layer disposed on the first electrode on a side opposite of the electrolyte, wherein the electrolyte and the stabilizer layer are formed of the same material (i.e., yttria stabilized zirconia). *See Exhibit B*, pgs. 2-3.

12. The presentation referred to in Paragraph 10 states that that "[q]ualitative measurements have confirmed improved flatness for cells containing stabilizing layers . . . ." *See Exhibit B*, pg. 5.

13. On or before August 1, 2002, I and Joseph M. Keller conceived that the solid-oxide fuel cell described in Paragraphs 11 and 12 may be formed using the method recited in Paragraph 4.

14. The presentation referred to in Paragraph 10 further discloses that the solid oxide fuel cell described in Paragraphs 11 and 12 also includes a second electrode (i.e., cathode) is disposed on a side of the electrolyte opposite the first electrode. *See Exhibit B*, pg. 2.

15. On or before August 1, 2002, Joseph M. Keller and I conceived that the solid-oxide fuel cell described in Paragraph 14 may be formed using the method recited in Paragraph 5.

16. I actually reduced to practice the invention included in Paragraphs 4 and 5 set forth above on or before August 1, 2002.

17. On or before August 1, 2002, I formed a ceramic assembly by performing the steps of: disposing an anode precursor on an electrolyte precursor; disposing a stabilizer precursor on the electrode precursor on a side opposite the electrolyte precursor to form a precursor assembly, wherein the electrolyte and the stabilizer were both formed of yttria stabilized zirconia; and sintering the precursor assembly to form the ceramic assembly comprising a stabilizer layer, electrode, and electrolyte. *See U.S. Patent Application No. 10/797,301, claim 1.*

18. On or before August 1, 2002, I formed a solid oxide fuel cell by performing the steps of: disposing an anode precursor on a solid electrolyte precursor; disposing a stabilizer precursor on the anode precursor on a side opposite the electrolyte precursor to form a precursor assembly, wherein the electrolyte and the stabilizer were both formed of yttria stabilized zirconia; disposing an cathode precursor on a side of the solid electrolyte precursor opposite the anode precursor; and sintering the precursor assembly to form the ceramic assembly comprising a stabilizer layer, first electrode, and electrolyte. *See U.S. Patent Application No. 10/797,301, claim 26.*

19. The presentation referred to in Paragraph 10 includes a photograph of the ceramic assembly/solid oxide fuel cell that I formed by performing the steps recited in Paragraphs 17 and 18, wherein the photograph shows, among other features, a screen printed stabilizer layer formed of yttria stabilized zirconia attached to a fuel cell frame. *See Exhibit B, pg. 8.*

20. The presentation referred to in Paragraph 10 illustrates the results of a test that I performed on a ceramic assembly/solid oxide fuel cell that I formed by performing the steps recited in Paragraphs 17 and 18, wherein the stabilizer layer was formed using tape punching. *See Exhibit B, pg. 7.*

21. The test referred to in Paragraph 20 was conducted to determine the amount of camber on a surface of the ceramic assembly/solid oxide fuel cell formed by performing the steps recited in Paragraphs 17 and 18. *See Exhibit B*, pg. 7.

22. The results of the test referred to in Paragraph 20 were obtained using a UBM Non-Contact Imaging System. *See Exhibit B*, pg. 7.

23. The test referred to in Paragraph 20 shows that a surface of the ceramic assembly had less than or equal to about 5.0 degrees camber as measured from a horizontal plane. *See Exhibit B*, pg. 7.

24. As a result of the test referred to in Paragraph 20, Joseph M. Keller and I recognized that the method recited in Paragraphs 17 and 18 successfully resulted in the formation of a ceramic assembly/solid oxide fuel cell with increased flatness relative to a standard solid-oxide fuel cell that did not include a stabilizer layer. *See Exhibit B*, pgs. 3, 7.

25. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on the information and belief are believed to be true, and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001, and that such willful false statements may jeopardize the validity of the application or any patent issued thereof.

Dated: \_\_\_\_\_

\_\_\_\_\_  
Robert J. Svoboda

*[Signature]*

**Record of Invention  
and**

**Request for Intellectual Property Investigation**

*copy*

File Number:	<u>DP 308347</u>
Product Code:	_____
For I.P. Office Use Only	

**INNOVATION CENTER  
FUEL CELL, SOFC**

This Record of Invention provides written documentation of your invention and initiates a process that may result in the filing of a patent application.

Delphi Unit: INNOVATION CENTER - FLINT Site/Location: Technical Center Flint

Product group this invention applies to: Solid Oxide Fuel Cells

Invention Title: Addition of Stabilizing Layer to Increase Solid Oxide Fuel Cell Flatness

**To disclose and record your invention, provide the following information:**

1. *To the best of your present knowledge, describe the background of your invention. Briefly describe the prior apparatus, material or process that is improved, replaced or most similar to your invention. What are the problems or shortcomings of the prior apparatus, material or process that are overcome by your invention?*

Flatness is an important aspect for the ceramic cell component of the solid oxide fuel cell system. Due to a shrinkage mismatch between the porous nickel and yttria stabilized zirconia anode and the dense yttria stabilized zirconia electrolyte the cell develops a camber during the sintering operation. Previously, the degree of camber was reduced slightly by a creep flattening process requiring the sintered cells to be fired at high temperature under ceramic weights. Creep flattening is time consuming and adds large amounts of energy due to the additional firing step required. Also, it only reduces camber by a limited amount.

2. *Describe your invention. Provide enough detail of the specific new features, components or steps that form the invention to enable a technical understanding of its content and novelty. Include a drawing with reference numbers keyed to your text description. Explain how your invention differs from and improves or solves the problems of the prior apparatus, material or process described above.*

The invention uses yttria stabilized zirconia or a material of similar shrinkage properties as a stabilizing layer on the anode side of the electrolyte-anode composite bi-layer. The similar shrinkage properties on each side of the anode will counter-balance the distortion caused by the shrinkage mismatch of the electrolyte and the anode.

Besides thermal expansion matching with the electrolyte, the stabilizing layer must account for these other critical properties:

- 1.) Sufficient transfer of fuel gas into the anode
- 2.) Electrical contact with the anode

Four solutions are proposed:

- 1.) An elastomer based yttria stabilized zirconia tape is applied to the anode side of the electrolyte-anode composite bi-layer. The tape is manufactured with an open hole or mesh pattern.
- 2.) A solvent-based yttria stabilized zirconia slurry is applied in an open hole or mesh pattern to the anode side of the electrolyte-anode composite bi-layer. The slurry can be applied using screenprinting, spraying, or other methods.
- 3.) An elastomer yttria stabilized zirconia tape containing fugitive material is applied to the anode side of the electrolyte-anode composite bi-layer. The fugitive material will burn off at temperatures below the sintering temperature causing voids in the fired ceramic.
- 4.) A solvent-based yttria stabilized zirconia slurry containing fugitive material is applied to the anode side of the electrolyte-anode composite bi-layer. The fugitive material will burn off at temperatures below the sintering temperature causing voids in the fired ceramic.

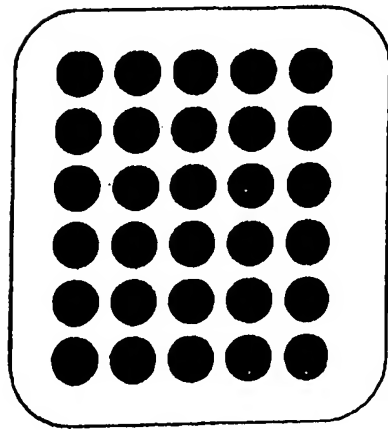
Key features of the open pattern design (1 and 2):

- 1.) The patterns must contain enough open area to allow sufficient gas flow to the anode
- 2.) Electrical contact must be made at the open areas

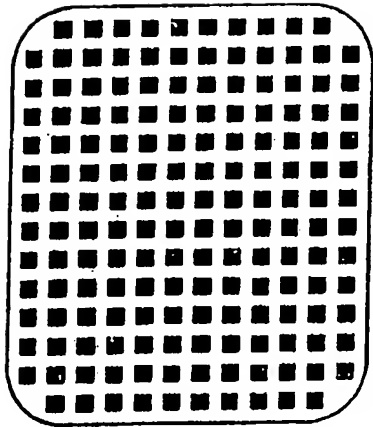
Key features of the fugitive material design (3 and 4):

- 1.) Porosity must be great enough to allow sufficient gas flow to the anode
- 2.) A conductive material (such as nickel) must be applied after firing. A solvent-based nickel slurry can be applied, diffusing into the pore structure making electrical contact to the anode layer. The nickel can be printed in an open pattern to allow for gas flow into the anode.





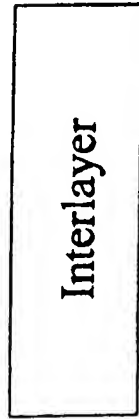
Hole Pattern



Mesh Pattern



Porous Material



Interlayer



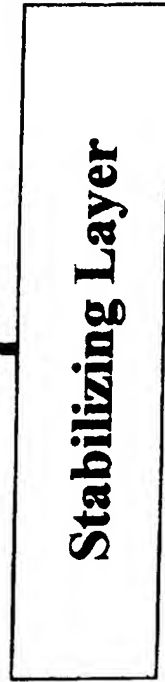
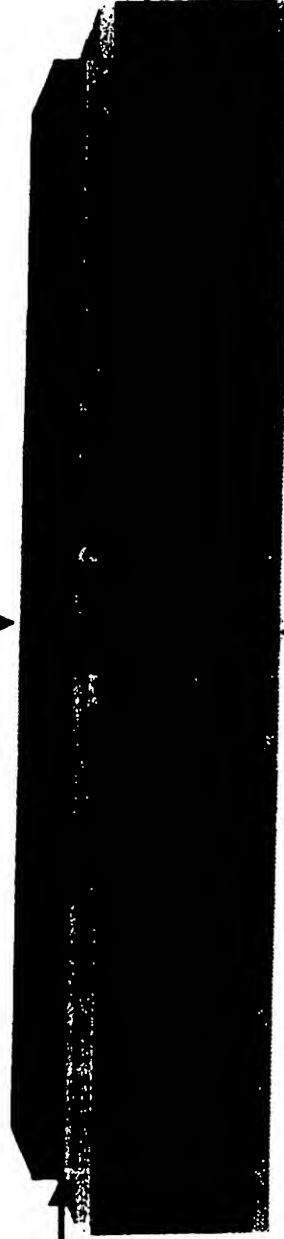
Cathode



Electrolyte



Anode



Stabilizing Layer

- 3.** Date this invention was first thought of: \_\_\_\_\_
- 4.** Attach a copy of the first written description and/or sketch of the invention. (preferably signed, dated & witnessed).
- 5.** Date this invention was or is expected to be disclosed outside of Delphi: no  
If disclosed, to whom: (customer, supplier, public, etc.) \_\_\_\_\_
- 6.** Date this invention was used or is committed to be used in production: \_\_\_\_\_
- 7.** Date this invention or a system including or using this invention was or will be offered for sale outside Delphi: \_\_\_\_\_
- 8.** Does this invention relate to a Government Contract? Yes ☐ No ☐  
If yes, identify the government Contract/Purchase Order No. \_\_\_\_\_

**Inventor #1**

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☐ No ☒ Contract ☐ Other

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2nd Level Manager: JIM ZICKELMAN Mail Code: 146-HEN-575 Telephone: [REDACTED]  
Contract Employer: \_\_\_\_\_ Telephone: \_\_\_\_\_  
(If applicable) (Area Code) + Number  
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Street City and State Zip Code

**Inventor #3**

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☐ No ☐ Contract ☐ Other  
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Contract Employer: \_\_\_\_\_ Telephone: \_\_\_\_\_  
(If applicable) (Area Code) + Number  
Contract Employer Address: \_\_\_\_\_  
Street City and State Zip Code

\*\* If there are more than three (3) inventors, copy this page as needed. \*\*

## **Guidelines for Description of Invention**

**Mechanical and Electrical Devices:** Include a detailed description and drawings which illustrate all essential elements of the invention and the environment in which it is used. The description should describe the structure and its operation key-numbered to the drawing(s). To the extent known, alternate embodiments should be described. In the case of systems and circuits, use accepted symbols or labeled blocks for standard components. A computer program or controlling algorithm, if important to the invention, should be provided in flow chart or similarly accepted format, and input/output signals should be identified and related to appropriate sensors/actuators.

**Chemical and Material Inventions:** Identify all essential materials (in chemical terms, not trade names) used and alternatives therefor. All significant variables needed to define the invention must be identified, quantified and discussed. Depending on the nature of the invention, such variables might include treatment/reaction times, temperature, pressure, concentration, particle shape/size, viscosity, crystal structure, phases, porosity, pH, density, tensile strength, polymer chain length, etc. Each variable should be quantified in terms of an operative range and a preferred embodiment, e.g. "The heat treatment is carried out between 100°C and 200°C (preferably 165°C)". The function/purpose of each variable should be described, including a statement as to what happens if the variable falls outside the operative range, e.g. "Component A serves as a plasticizer for Component B. Below 100°C, Component A will not mix with Component B, and above 200°C it evaporates." Finally, a recipe for at least one detailed working example should be provided. Preferably, several such examples will be provided covering the full range of the significant variables used to define the invention.

**Processes:** Include a schematic of the components monitored, controlled and/or created by the process and a flow chart of the process illustrating the steps of the process at a level from which one skilled in the art can understand and implement the process. The description should set forth the operation of the process describing each step of the flow chart and its relationship or interaction with the components monitored, controlled and/or created by the process.

## **Protecting Your Invention**

When a new idea comes to you, immediately make a drawing or sketch of it and write a brief description sufficient to make your invention clear to others working in the same field. Show the drawing and description to two of your associates who are not co-inventors and make sure they understand your invention. You and your co-inventors, and your associates as witnesses, should sign and date the drawing and description. The date used must be the date of the signature. The witnesses should state above their signatures that the drawing and description were reviewed and understood by them. Include the documents with your Record of Invention.

Be aware that patent rights may be lost irrevocably by action of law, in spite of timely documentation and submission of a Record of Invention, through actions taken by you or others prior to an actual patent application being filed in the appropriate government Patent Office. Such actions may include: (a) publishing the invention, (b) using the invention in public or commercially in our plants, (c) disclosing or offering to sell an item incorporating the invention outside Delphi or (d) obtaining a quote for such an item from a supplier. Please submit your Record of Invention without delay when you think of an invention and report any past or planned disclosure of the invention outside Delphi or commercial use of the invention so appropriate steps may be taken to avoid loss of patent rights.

## Authorization

**I hereby assign this invention to Delphi Technologies, Inc. and authorize Delphi Technologies, Inc. to file a patent application on my behalf.**

INVENTOR - SIGNATURE Robert J. Svoboda DATE [REDACTED]  
(ALSO, PRINT NAME)

W. A. G. L. HASKELL SIMPSON [REDACTED]  
INVENTOR - SIGNATURE (ALSO, PRINT NAME) DATE



INVENTOR - SIGNATURE JOSEPH M. KELLER DATE                     

Inventor verifies this date.

Jack Tubbs,

**This invention was reviewed and understood by the witnesses below:**

David P. Wallace II David P. Wallace II [REDACTED]  
1st WITNESS - SIGNATURE (ALSO, PRINT NAME) /DATE

2nd WITNESS - SIGNATURE
 CHARLES D OAKLEY
 DATE

# **DELPHI**

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**Addition of Stabilizing Layer to Increase Solid Oxide Fuel Cell Flatness**  
**Invention No. DP-308342**

**Robert Svoboda**  
**Haskell Simpkins**  
**Joe Keller**

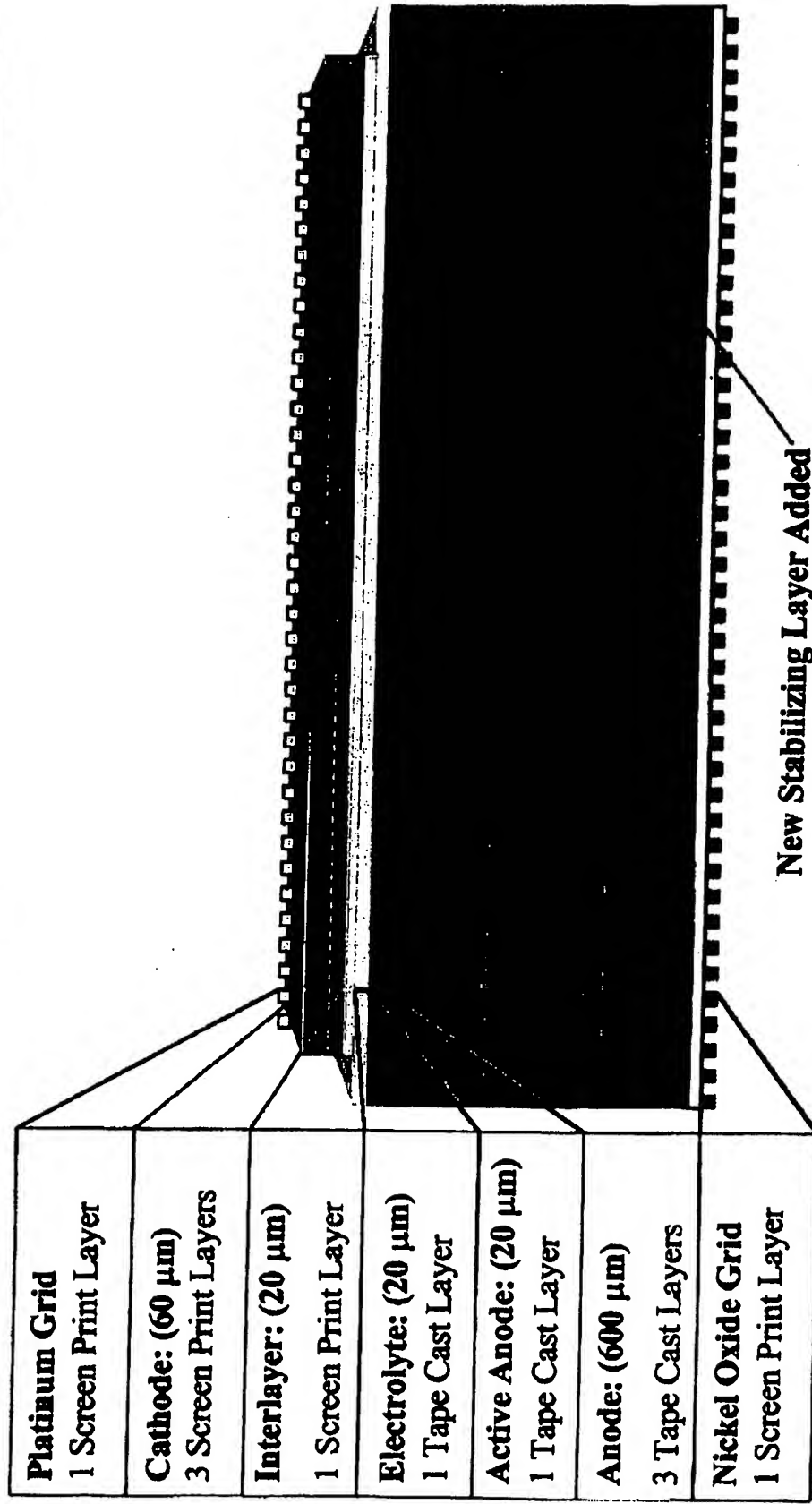
**08/01/02**

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**Energy & Engine Management Systems**

**DELPHI**

Solid Oxide Fuel Cell



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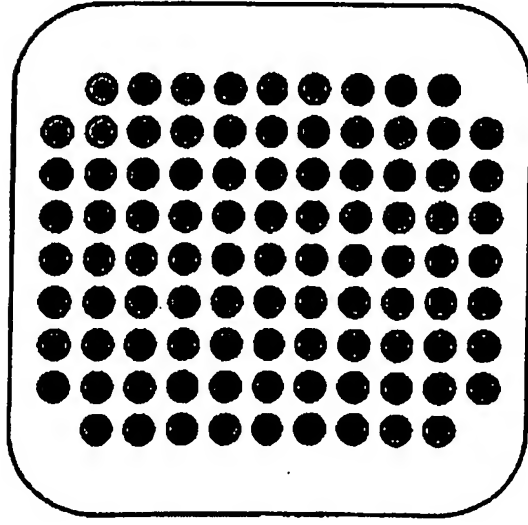
Energy & Engine Management Systems

- ◆ Yttria stabilized zirconia placed on both sides of cells
- ◆ Design Criteria for “stabilizing” zirconia layer
  - Electrical impedance must not be significantly increased
  - Gas flow through the anode must not be significantly decreased
- ◆ Improvements in cell quality
  - Increased mechanical strength and durability
  - Reduced cell warping
  - Enhanced sealing

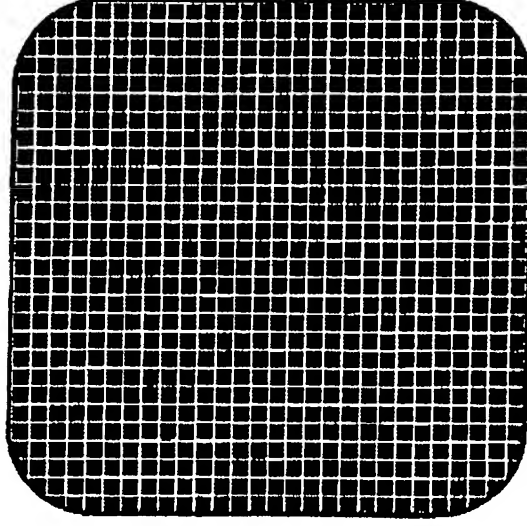


**DELPHI**

Stabilizing Layer Designs



Hole Punched Tape



Screen Print



Porous YSZ Layer

# **DELPHI**

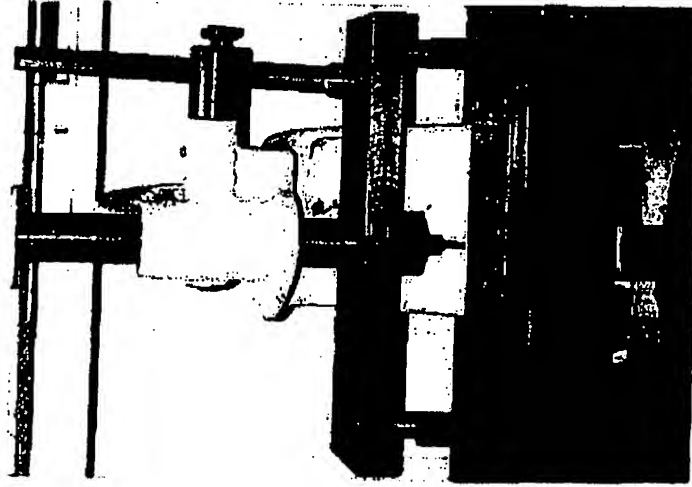
## **Current State**

- ◆ Cells have been manufactured using tape punching, screen printing, and porous tape methods methods
- ◆ Quantitative measurements have confirmed improved flatness for cells containing stabilizing layers that were fabricated using tape punching and screen printing methods
- ◆ Cell with screen printed stabilizing layer successfully sealed to frame.

**DELPHI**

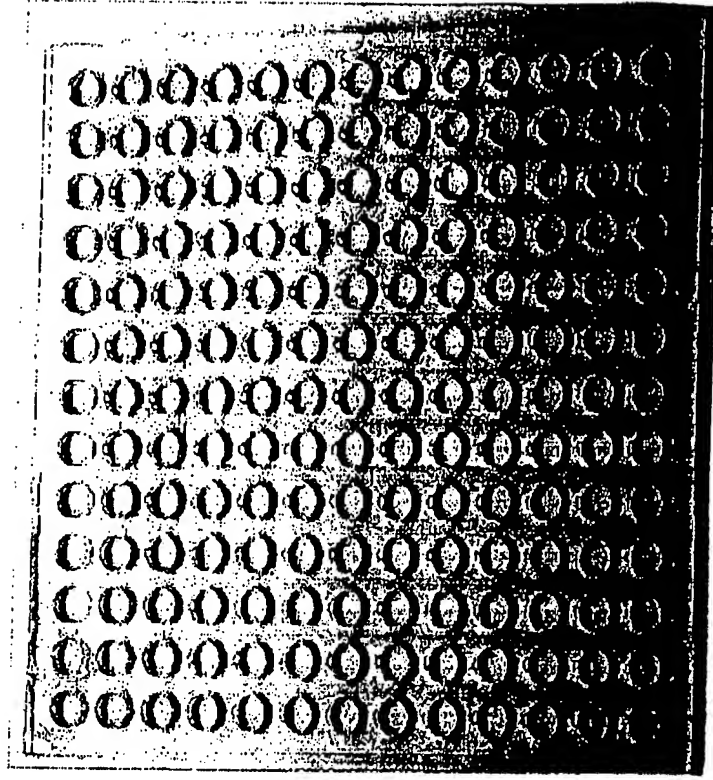
# Tape Punching System

Hole Press



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Hole Fixture



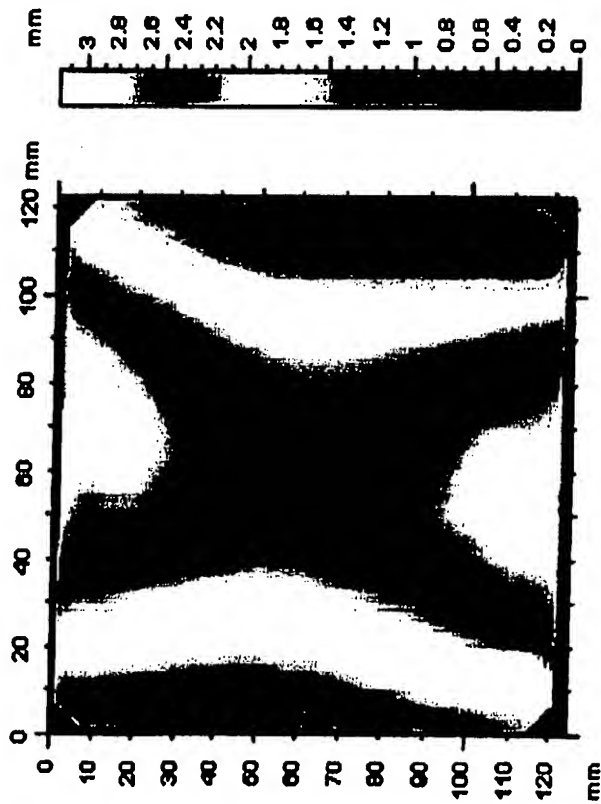
*Energy & Engine Management Systems*

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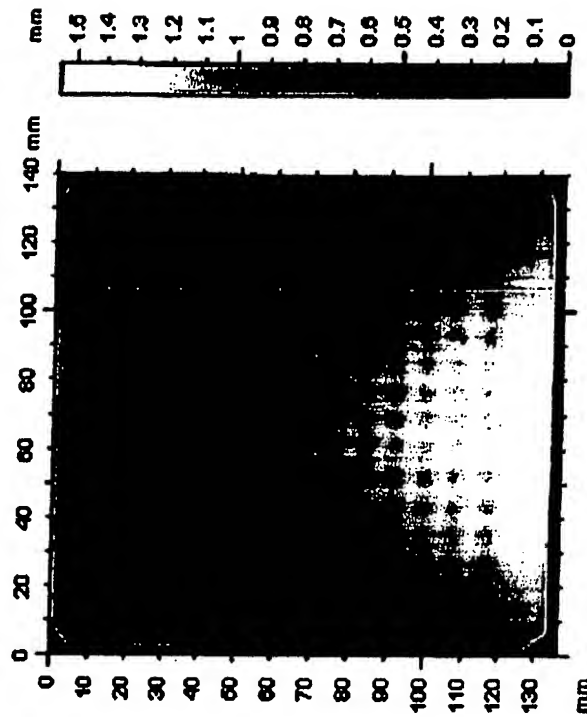
## Tape Punched Cell Flatness Measurement Comparison

Solid oxide fuel cell camber of standard cell and a cell containing a punched tape - cast stabilizing layer as measured on a UBM Noncontact Imaging System.

Standard Cell



Cell with stabilizing layer



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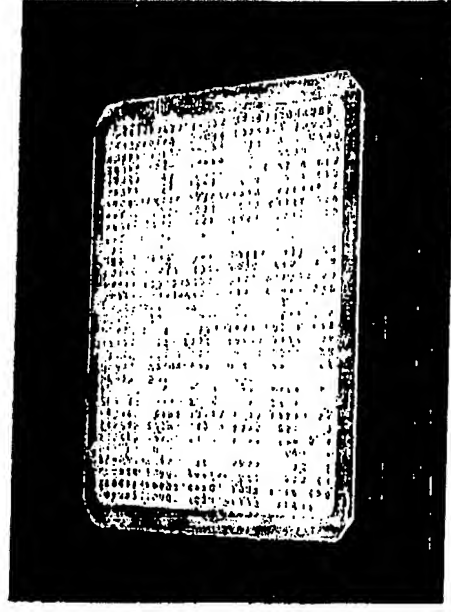
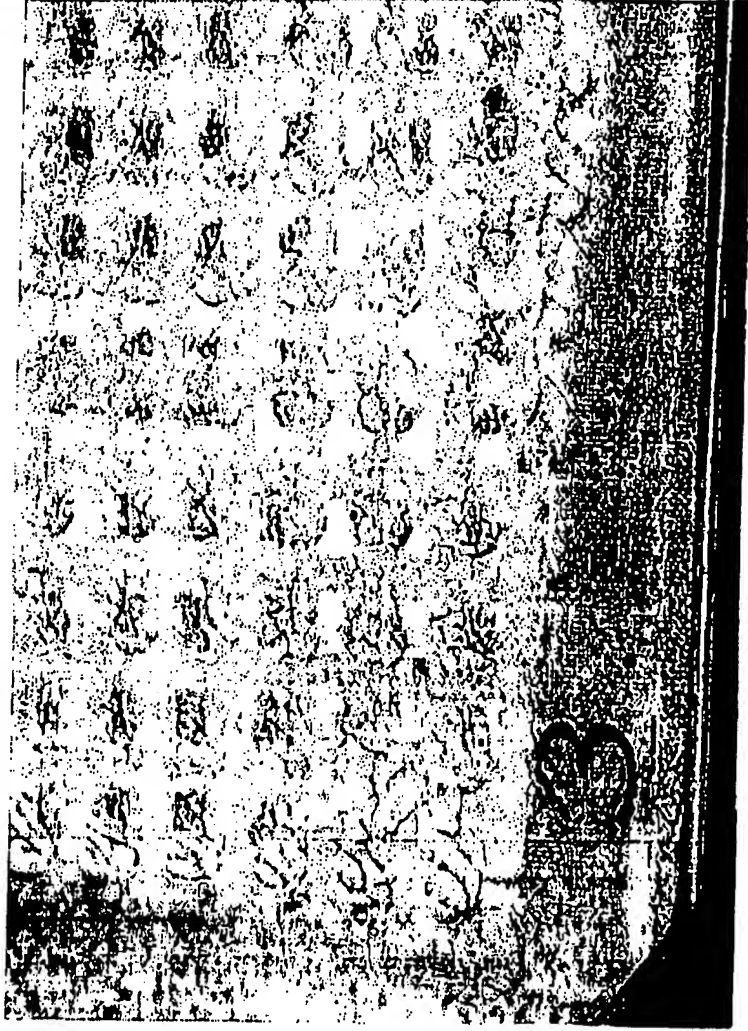
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# **DELPHI**

## **Screen Printed Cell Sealed Into Frame**

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Yttria stabilized zirconia screen printed grid underneath  
nickel grid (below)



Yttria stabilized zirconia screen  
printed cell attached to frame  
(above)

# Exhibit C

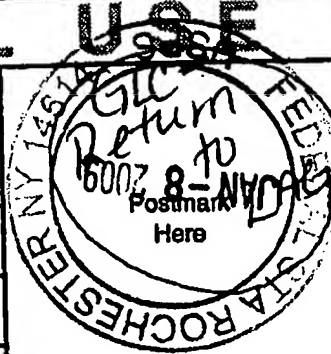
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See Reverse for Instructions

# Exhibit D



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48473

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**X** *Mark Svoboda* ☐ Agent ☐ Addressee

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**MARK SVOBODA** C. Date of Delivery

D. Is delivery address different from item 1? ☐ Yes  
If YES, enter delivery address below: ☐ No

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